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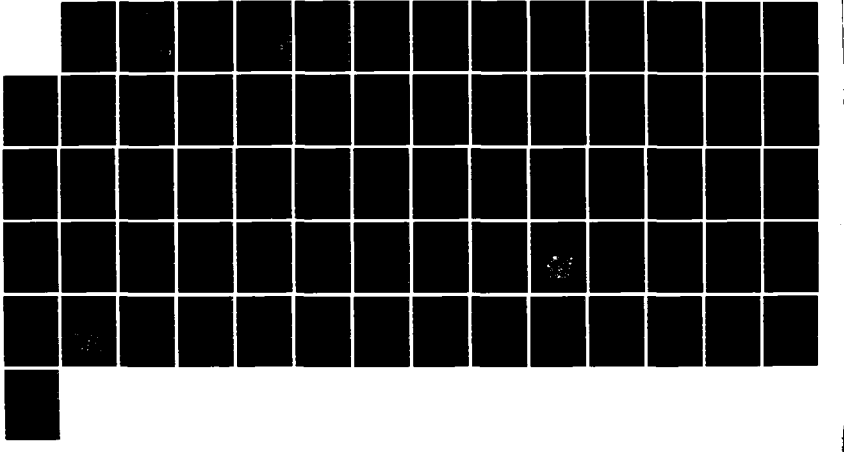

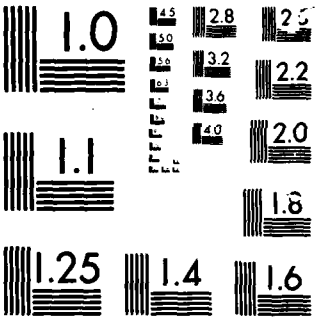

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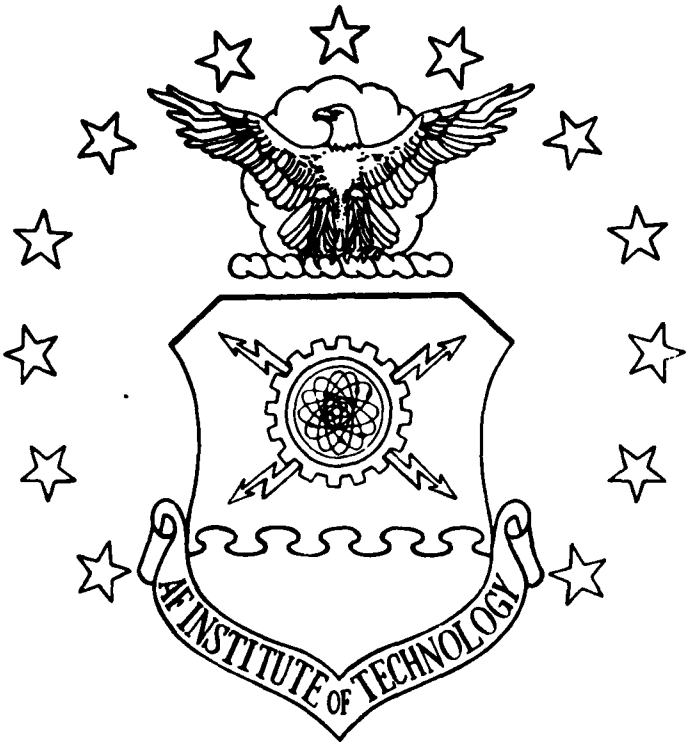

DETECTION OF ALPHA PARTICLE INTERACTIONS USING THE TRACK ETCH TECHNIQUE

THESIS

Robert C. Myers
Captain, USAF

AFIT/GNE/ENP/85M-16

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THESIS

Presented to the Faculty of the School of Engineering
of the Air Force Institute of Technology
Air University
In Partial Fulfillment of the
Requirements for the Degree of
Master of Science in Nuclear Engineering

Robert C. Myers, B.S., M.S.
Captain, USAF

March 1985

AFIT/GNE/ENP/85M-16

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Dean, School of Engineering

PREFACE

The purpose of this study was to compile a data base of experimental procedures and methodologies to investigate alpha particle interactions using the track etch technique. Specifically, the $^{10}B(n,\alpha)^{7}Li$ reaction was investigated because of its applicability to both civilian and military environmental studies.

This report is limited in scope to: 1) compilation of procedural methods for investigation of alpha particle interactions using the track etch technique, 2) presentation of track formation theory, and 3) qualitatively report observations obtained from samples processed using these procedures. This work provides a basic understanding of the track etch technique and how it can be applied to many areas of alpha particle research.

I would like to thank my advisor, Major John Prince, and Dr. George John of the Air Force Institute of Technology for their intuitive guidance critical to the completion of this study. Appreciation is also expressed to Dr. Philip Jenkins of Mound Laboratory, Mary Parkhurst and Larry Brackenbush of Pacific Northwest Laboratory, and the Air Force Technical Applications Center (AFTAC) for providing the track etch materials. Special thanks to Mr. Dale Hankins of the Lawrence Livermore National Laboratory for providing current information of the electrochemical etch (ECE) process. Finally, I wish to thank my wife, Donna, whose inspiration and typing skills made completion of this thesis possible.

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Abstract

In this study I have developed and set up experimental procedures and methodologies for detection of low energy alpha particles using the track etch technique. Three track etch materials, lexan polycarbonate, cellulose nitrate, and CR-39, were exposed to various energy alpha particles. The exposure resulted in the formation of atomic scale damage tracks within each material. Because the damage tracks were on an atomic scale in size, they could not be optically seen unless enhanced or enlarged by some physical means. Three methods of enhancement were performed on the exposed track etch materials. These methods were chemical etching, electrochemical etching, and a combination of the two. The results indicted that all three etching methods provided adequate enhancement of the alpha damage tracks, thereby showing that low energy charged particles can be detected using the track etch technique.

DETECTION OF ALPHA PARTICLE INTERACTIONS USING THE TRACK ETCH TECHNIQUE

I. Introduction

Background

In 1959, Silk and Barnes first investigated nuclear particles by observing their interactions with track etch materials. Since that time, track etch materials have been used in numerous fields of endeavor. These include nuclear science, astrophysics, geology, archeology, lunar science and meteoritics (1:3). The major emphasis of the early studies was the investigation of high atomic number, heavily charged particles (i.e. fission fragments and high Z, high energy cosmic rays). Recently the analysis of low Z and low energy particles (i.e. alphas, protons and neutrons) has grown in importance. This growth in importance has been caused by expansion in the nuclear industry. Because of the increased use of radioactive materials and nuclear power generation, development of a more sensitive personal radiation monitoring system was needed. New track etch materials and procedures provide this capability, allowing low energy alphas, protons and neutrons to be detected and measured.

Objective

The objective of this study was to compile and perform track etch procedures capable of detecting alpha particle interactions within track etch materials. Specifically, the $^{10}B(n,\alpha)^{7}Li$ reaction was investigated because of its potential use in environmental studies at the McClellan Central Laboratory (MCL) of the Air Force Technical

Applications Center (AFTAC). Direct alpha particle interactions from ^{241}Am and environmental radon were also investigated and are presented with the $^{10}B(n,\alpha)^{7}Li$ results.

Scope

The study is limited in scope to: 1) compilation of procedural methods presently used to investigate alpha particle interactions in track etch materials; 2) presentation of track formation theory; and 3) discussion of the results obtained from samples using these track etch procedures. No attempt is made to give quantitative values to the number of interactions observed within the track etch materials.

Approach

This study was performed by a four-step process. A literature search of past and present track etch techniques and theories was performed to form a data base from which information was drawn. The most promising procedures and track etch materials for detection of alpha particles were chosen from this data base. Equipment and materials needed to perform these procedures were either designed and constructed, or acquired from outside sources. The track etch materials were then exposed, etched, and analyzed following the chosen experimental methods.

Presentation

A general background on boron and the $^{10}B(n,\alpha)^{7}Li$ reaction is presented in Chapter II. A generic description of track etch materials, their uses, sensitivity, and problem areas is provided in Chapter III.

A discussion of track formation and etch theory is presented in Chapter IV. The experimental equipment used in the study is described in Chapter V, followed by a description of the experimental procedures performed and the results obtained in Chapter VI. A summary and recommendations is presented in Chapter VII.

II. Boron and the $^{10}B(n,\alpha)^{7}Li$ Reaction

Boron is a nonmetal found as a salt in the ocean and dry lake beds of ancient inland seas. It is the first and lightest member of the group IIIA elements of the periodic table having an atomic number of 5 and an atomic weight of 10.811. Boron exists as two naturally occurring isotopes: ^{10}B (19.61%) and ^{11}B (80.39%). Its properties include outstanding lightness, stiffness, hardness, resistance to stretching, and excellent thermal stability. For this reason, filaments of boron are combined with metals and plastics to form composites which are superior in strength to either material alone (2:48). Boron also plays an important role in the detection and shielding of neutrons.

The ^{10}B isotope has a thermal neutron capture cross section of 3837 barns (10^{-24} cm^2) and therefore, provides an excellent shielding material within nuclear reactors (3:2). The capture of a thermal neutron by a ^{10}B atom results in the release of a low energy alpha particle, a lithium atom, and a gamma ray.

$$^{10}_{5}B + ^{1}_{0}n \longrightarrow ^{7}_{4}Li + ^{4}_{2}He + Q + \gamma$$

The energies of the particles emitted are found using the mass-energy balance equations below (4:61-64).

$$KE_{Li} = \frac{M_{Li}}{m_{\alpha} + M_{Li}} \left(Q - \gamma \right) \qquad (1)$$

$$KE_{\alpha} = (Q - \gamma) - KE_{Li} \qquad (2)$$

where

KE_{Li} is the kinetic energy of the lithium atom

KE_{α} is the kinetic energy of the alpha particle

γ is the energy of the gamma ray emitted

M_{Li} is the atomic mass of lithium

m_{α} is the atomic mass of the alpha

Q is the mass energy decrement value

From these formulas the following particle energies are calculated:

$Q = 2.80$ Mev

$\gamma = 0.49$ Mev (measured)

$^{7}_{3}Li = 0.84$ Mev

$^{4}_{2}He = 1.47$ Mev

Because the particles emitted from the reaction are potential radiation hazards, shielding of these particles must be considered. Shielding requirements for the alpha particle and lithium atom can be determined using the specific energy loss relationship. Specific energy loss, the rate at which a particle loses energy by interactions with a material, is related to the energy per nucleon and the charge of the particle. As the energy per nucleon decreases, velocity of the particle decreases and specific energy loss increases. If two particles are of equal energy, but different charge (Z), then the one with the higher charge will have the greater specific energy loss (1:24, 5:41-46). The energy per nucleon of the alpha particle and lithium atom are 0.3675 MeV and 0.12 MeV respectively. Since the lithium atom has less energy per

nucleon and a higher charge (3 vs 2), it will be stopped in a shorter distance than the alpha particle. Using Figure 1, the range of a 1.47 MeV alpha particle in air is only 0.75 centimeters (5:46). This short range makes shielding trivial, but also makes the reaction highly useful in other areas of study.

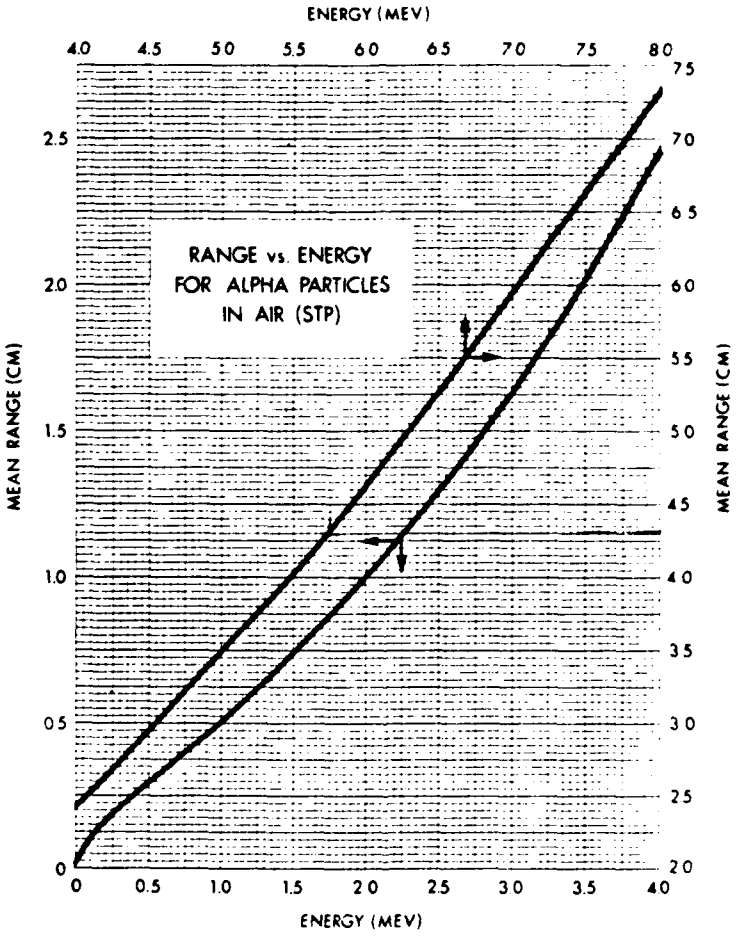

Fig 1. Range vs Energy Plot for Alpha Particles in Air (STP)

The $^{10}B(n,\alpha)^{7}Li$ reaction can be used for the detection of neutrons and the treatment of cancer (i.e. brain tumors, breast, uterus and prostate cancers) (6:50-51). Neutrons are detected by filling a proportional counter with gaseous boron trifluoride. Upon entering the boron trifluoride, neutrons are captured by the ^{10}B atoms and release alpha particles, triggering a response in the detector. Of greater importance is the use of ^{10}B to fight cancer. In this process a ^{10}B-loaded compound is injected into the patient and is preferentially absorbed by the cancer cells. The area is then bombarded with a beam of thermal neutrons and the $^{10}B(n,\alpha)^{7}Li$ reaction takes place. Since the alpha particles and lithium atoms are of such low energy (1.47 MeV, 0.84 MeV respectively), destruction of cell tissue is localized to the cancerous region. Thus, tumors and cancers which have limited surgical success can be effectively destroyed by localized radiation.

III. Track Etch Materials

Track Etch Materials

Track etch materials are dielectric solids which form damage tracks under the passage of heavily ionized nuclear particles. These tracks are on atomic scale in size and must be enlarged by some physical means. The materials can be crystalline or polymeric in structure and organic or inorganic in composition (1:1-3). The track etch materials chosen for this study were organic polymers. They included lexan polycarbonate, cellulose nitrate, and CR-39.

Material Sensitivities

Lexan polycarbonate, although "low" in alpha particle sensitivity, was chosen because of its present use at MCL as a tool for environmental studies. Cellulose nitrate and CR-39 were selected because of their "high" sensitivity to alpha particles. *Figure 2 shows* the sensitivity of lexan polycarbonate and cellulose nitrate as a function of energy per nucleon and particle velocity (1:18).

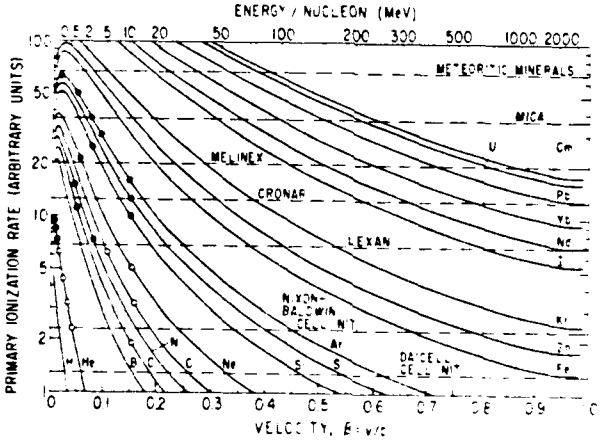

Fig 2. Plot of Track Etch Material Sensitivities vs Energy Nucleon and Particle Velocity for Different Charged Particles

Figure 2 shows that lexan polycarbonate will not register alpha particles with energies greater than 0.5 MeV per nucleon or 2.0 MeV per alpha particle. The particular brand of cellulose nitrate used will not register alphas greater than 5.5 MeV (7). CR-39 is a relatively new material, but has exhibited sensitivity to protons and neutrons of 10 keV and 20 keV respectively (8).

Sensitivity of these organic polymers may vary drastically. Differences may exist from batch to batch, in the same batch, or on the same sheet. Material sensitivity is influenced by: 1) manufacturer's process; 2) material curing time; 3) material curing temperature; 4) concentration of chemicals and impurities within the polymer; 5) material molding technique; 6) molding materials; and 7) material storage (9:74-82). To eliminate the fluctuation in material sensitivity, these materials must be manufactured under stringent guidelines. However, this is generally not the case. Cellulose nitrate and CR-39 are not specifically manufactured for track etch use. Their success as track etch materials is of little importance to the manufacturer.

Uses of the Materials

Cellulose nitrate is an integral component of explosives, plastics and monopropellants. It is a highly volatile substance which burns very rapidly when exposed to heat or flame. Different manufacturing processes produce various amounts of impurities within it. These impurities affect the sensitivity of the material to radiation damage (7).

CR-39 is a commercial plastic used in the fabrication of sunglasses and prescription lenses. Because different manufacturers supply the material, none is exactly the same in composition. Therefore, the sensitivity of CR-39 to radiation damage is quite variable (10).

IV. Track Formation and Enhancement

Track Etch Damage Mechanisms

As previously stated, track etch materials are dielectric solids which form damage tracks on an atomic scale as a result of the passage of heavily ionized nuclear particles. Although a simple statement, the formation of the damage tracks is much more complex. At present, no one theory fully relates or explains the damage mechanism in the organic and inorganic solids. Theories for inorganic materials are different from those for organic ones (1:31). Several of the detailed theories are found in the Appendix.

General Description of Track Formation

As a fast atom (particle) of atomic number Z passes through a solid, it becomes a positively charged ion. This ion is formed when electrons of the moving atom are "stripped" away by interactions with charges in the surrounding solid. The most prominent damage mechanism between the ion and atoms of the solid is electrical force. As a result of this electrical force, one of two events occurs: 1) the electrons within the atoms of the solid are excited to higher energy levels; or 2) the electrons within the atoms of the solid are ejected from their orbital shells by coulomb attraction (1:24-25, 5:46). The ejected electrons are called delta rays. The delta rays may possess enough energy to produce further excitation and ionization within the atoms of the solid.

The first set of events is primary ionization and excitation. The

delta ray interactions are secondary ionization and excitation. Primary ionization and excitation damage is localized to the path of the ion. Secondary ionization and excitation damage is distributed over a larger radial distance from this central path. Primary and secondary ionization and excitation can cause charge centers to develop in both organic and inorganic solids. They may also break long chain molecules and form free radicals in the organic polymers (1:24-25).

As the ion slows in the solid, it reacquires orbital electrons and becomes less ionized. When it reaches approximately 50 keV per atomic mass unit (amu) or less, direct atomic collisions with the atoms of the solid become the dominant damage mechanism. This direct atomic interaction can create displaced atoms and vacancies within the track etch materials (1:24). Figure 3 pictorially shows the track formation mechanism in crystalline and polymeric track etch materials.

Damage Track Enhancement

Because the damage tracks are on atomic scale, they cannot be seen optically unless enhanced or enlarged by some physical means. The primary methods of enhancing damage tracks are chemical etching (CE), electrochemical etching (ECE), and their combination. Each method is described in detail in the next three sections.

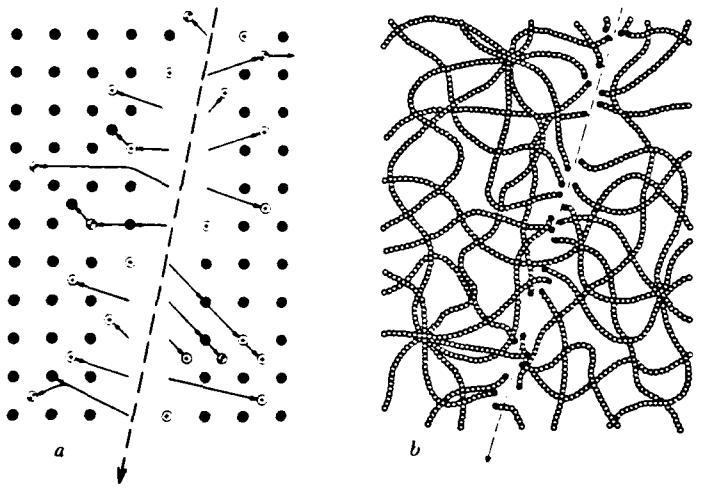

Fig 3. General Track Formation Mechanism in Crystalline and Polymeric Track Etch Materials. (a) Vacant lattice sites and interstitial ions or atoms in a crystal and (b) new chain ends in polymers.

Chemical Etching

The simplest method of damage track enhancement is chemical etching. In this procedure the track etch material is bathed in a chemical reagent that slowly dissolves it. The rate at which the material dissolves is related to the material's chemical composition and structure. The ionized particle has created a region in which the chemical composition and structure are changed. The change in material properties creates a dissimilarity in the bulk material etch rate, V_B, and the damage region etch rate, V_T. The etch rates V_B and V_T are critical to enhancement of the damage track. V_T must always be greater than V_B for track enhancement, by chemical etching, to be successful.

For track enhancement to work, (assuming V_T is constant along the track and V_B is constant and isotropic) the particle incident on the dielectric material must have entered at an angle greater than θ, where $\theta = \arcsin V_B/V_T$. If the particle enters at an angle less than θ, the normal component of V_T is smaller than V_B and the surface of the bulk material etches away faster than the damaged material as shown in Figure 4. The dimensions of the hole are governed by the amount of time the material is in contact with the chemical reagent. By knowing the dimensions and angles of the holes, estimates of the incident particle energy can be calculated.

Factors Affecting Chemical Etch Rate. Factors which affect the etch rate and thus the size of the damage tracks are: 1) etchant concentration; 2) etchant viscosity; 3) etchant temperature; and 4) etching time. Increasing the etchant concentration increases the overall material etch rates. This is as it should be and can be compared to an acid that is used to dissolve a metal disc. If the concentration of the acid is increased, so does the rate at which the disc dissolves. Reducing the etchant viscosity also increases the material etch rates. As the viscosity of the etchant is reduced, it becomes thinner. Because it is thinner, fresh etchant is circulated on the material surface at a higher rate. Fresh etchant dissolves the material faster than "old" etchant (i.e. etchant containing dissolved track etch material), thereby increasing the etch rates. Increasing the etchant temperature also increases the material etch rates. As the track etch material is heated, the chemical bonds between its molecules become weaker. The weaker bonds are more susceptible to attack and

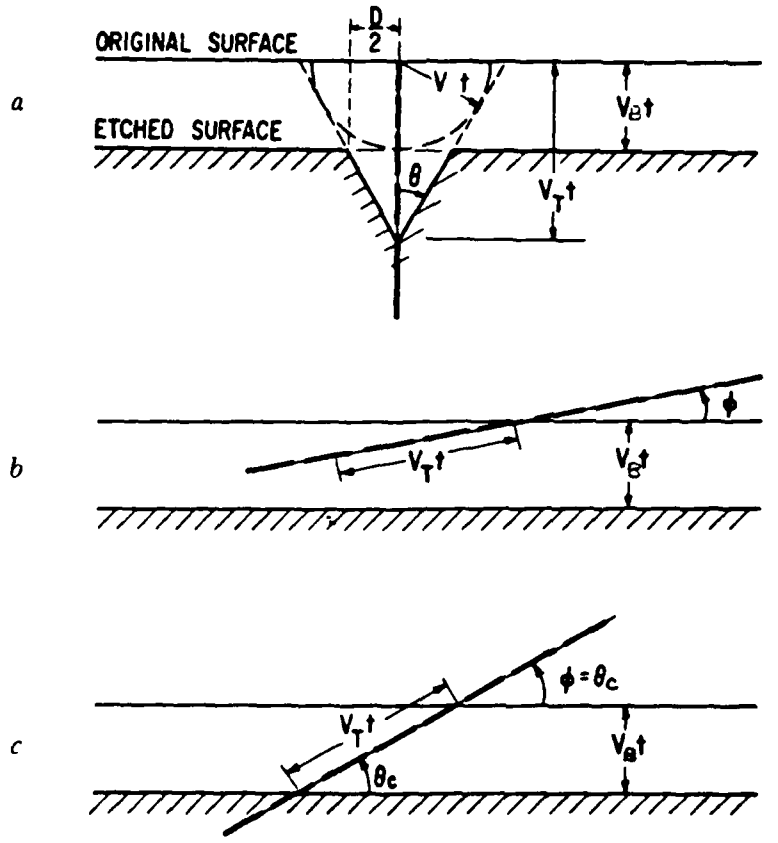

Fig 4. Track Enhancement by Chemical Etching

(a) track pit shape is determined by the general rate of attack V_B and the preferential attack rate V_T along the damage trail; (b) for angle of incidence less than arcsin (V_B/V_T), the surface is removed at a greater rate than the normal component of V_T and therefore no track is observed; (c) arcsin (V_B/V_T) is the critical angle θ_c, above which tracks are registered (1:58).

therefore, the etch rates increase. Also, as the etchant is heated, the ions within it become more active. This increased activity increases the rate of etchant circulation on the material surface. The increase in etchant circulation results in faster etch rates. Etch time is related to all three factors. Etching the material in a high temperature, high concentration etchant will lower the etch time significantly. For example, if a damage track of one micron is desired, use of a low concentration, room temperature etchant may require 15-20 hours etch time. Doubling the concentration and raising the etchant temperature to 60°or 70°C could reduce the etch time to less than five hours (11:242).

Problems with Chemical Etching. Care must be taken to choose the optimum etch conditions when performing the chemical etch method. Excessive etching can completely remove the damage tracks. Once the chemical etchant has completely dissolved the damaged material, only bulk material remains and the etch rate V_T now becomes V_B. At this stage, a well defined hole surrounded by bulk material exists in the track etch material, as shown in Figure 5(a).

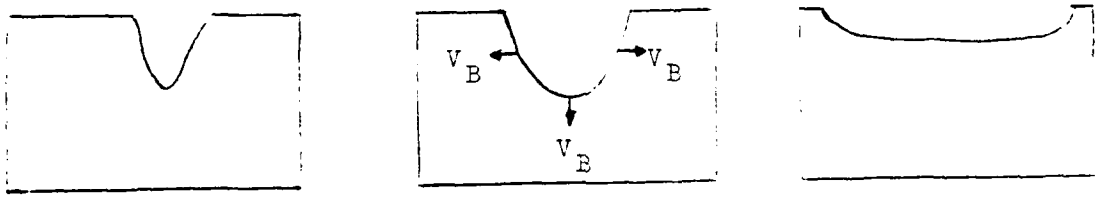

Fig 5. Results of Excessive Chemical Etching

As the etching process continues the hole becomes shallow and less defined. This is caused because the diameter of the hole etches out at a rate twice that at which the bottom is etched down, as shown in Figure 5(b). After further exposure the hole becomes so shallow that any contrast in depth cannot be observed under the microscope (1:50-60). Figure 5(c) illustrates this step. The hole has therefore been completely removed or etched away.

Electrochemical Etching

Electrochemical etching (ECE) is similar to chemical etching except that an AC electric voltage is applied to the dielectric solid while it is chemically etched. Figure 6 shows a schematic of a typical ECE apparatus.

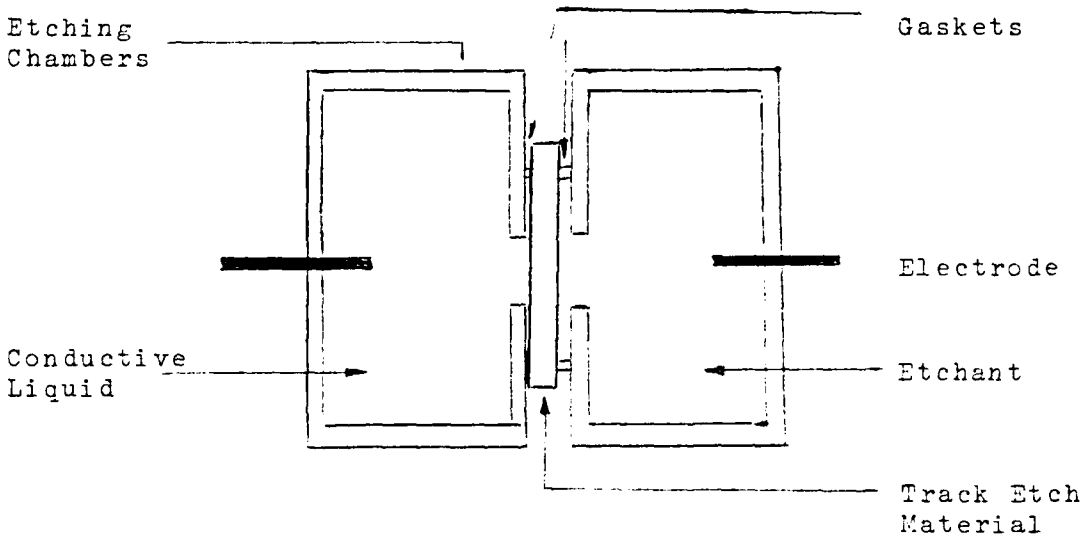

Fig 6. Typical Electrochemical Etching Apparatus

The exposed track etch material is positioned between two reservoirs of liquid. One reservoir contains the etchant and the second a conductive liquid. An AC voltage of a specific frequency is then applied to electrodes embedded in the reservoirs. This induces an electric field between the front and back face of the dielectric track etch material (12:414-424). Enhancement of the damage track by this procedure is a two-step process. First the chemical etchant etches the damaged region forming the conical hole or pit as previously described. As the pit deepens, the distance between the front and back face of the track etch material decreases. This causes an increase in the electric field at the tip of the pit region. As the electric field increases, free ions in the etchant are accelerated, by electrical attraction, into the dielectric solid. These ions impact the surface of the pit and impart energy an hence, force to it. The value of this force, as defined in the Georgia Institute of Technology Annual Progress Report on ECE, is found using the following formula: (13:37)

$$\vec{F} = Zi(e)\vec{E} \qquad (4)$$

where

$\vec{F}$ is the induced force

Zi is the elementary charge of the etchant ions

e is the unit electric charge

$\vec{E}$ is the electric field strength at the ions location

This imparted force creates mechanical stresses and strains in the region of impact. These stresses and strains damage the structural composition of the dielectric solid. The high electric field can produce other damage mechanisms which affect the structural composition of the dielectric solid. The high electrical stresses themselves can cause the bonds between the molecules of the dielectric to weaken and even break, making them more susceptable to the etchant. The electric field may produce dielectric or Joule heating in the tip of the track. As described earlier, a heated etchant has a faster etch rate. The high electric field may also cause the etchant in the tip of the track to vaporize. The increased pressure can cause the development of submicro cracks in the tip region. Finally, the mechanical stresses and strains may cause mechanical fatigue cracking or localized electrochemical breakdown. All of these mechanisms damage the composition of the polymer, thereby forming new damaged material (12:419). The chemical etchant attacks this newly damaged material creating further indentations in the new pit area. As the new indentations are formed, the electric field in these regions is increased. The etchant ions are again accelerated into the dielectric solid. This process, called "treeing", continues until the electric field is removed (12:414-424). Figure 7 illustrates this two-step process of pit formation and treeing. Because the treeing process completely destroys the primary damage track (hole), only the number of interactions which occur in the material and not their energies can be determined.

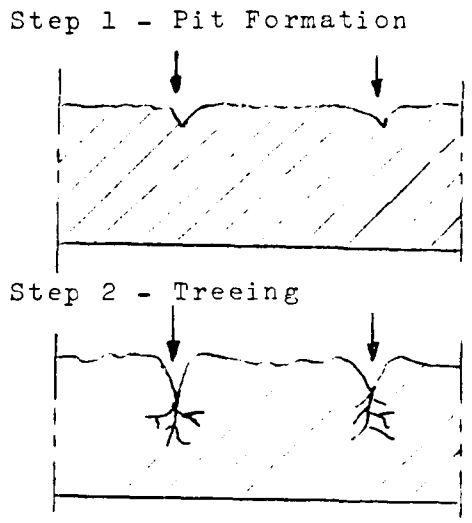

Fig 7. Pit Formation and Treeing by Electrochemical Etching

Factors Affecting Electrochemical Etching. Factors which affect ECE are: 1) etchant concentration; 2) etchant viscosity and conductivity; 3) etchant temperature; 4) magnitude of applied AC voltage; 5) frequency of applied AC voltage; and 6) etching time. As previously described, increasing the etchant's concentration, lowering its viscosity, and raising its temperature all result in faster etch rates. Increasing the voltage, frequency and etchant conductivity increase the treeing rate. As increased voltage is applied, free ions of the etchant obtain and impart greater energy to the pit of the damage track. This increase in deposited energy causes the formation of larger damage regions, or trees, in the pit area. Increasing the voltage frequency increases the rate of ion impaction on the material surface. The increase in impaction rate leads to an increase in energy

deposited in the pit region. The increase in deposited energy causes the formation of larger damage regions in the pit area, as did increasing the voltage. Increasing the etchant conductivity increases the number of free ions in the etchant. The increased number of of ions allows more ions to impact in the pit. Therefore, increasing the etchant conductivity is similar to increasing the voltage frequency. All three factors can significantly affect the treeing rate.

Problems with Electrochemical Etching. Two problems exist when ECE is used alone. Because a hole or pit is needed before treeing can begin, any imperfection on the surface of the dielectric could produce false treeing. This will give an incorrect reading (too high) as to the number of direct radiation interactions in the track etch material. Figure 8 shows this problem schematically.

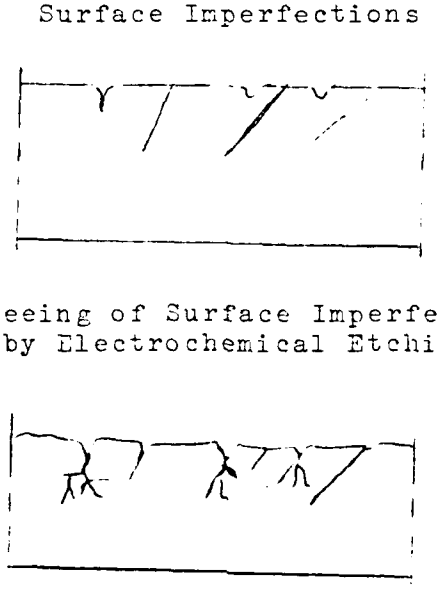

Fig 8. False Treeing in Electrochemical Etching

The second problem is similar in nature. If the chemical etchant is too low in concentration, or temperature, pit formation may take longer to begin than does the treeing process. When this occurs, areas damaged by higher energy particles may not be registered because of overshadowing by adjacent low energy damage regions. Again this will give an incorrect reading (too low) as to the number of direct radiation interactions in the material (12:414-424).

Chemical Etch and Electrochemical Etch Combination

In this procedure the irradiated dielectric solid is pre-etched in a chemical etch bath and then electrochemically etched. The chemical pre-etch insures that surface imperfections are removed and that an adequate pit is formed for the ECE process. ECE is then used to enlarge the damage tracks and insure that none are lost due to incorrect time exposure to the chemical etchant.

Etching Solution

Choosing one etchant which optimizes the etching rate in several different materials is not an easy task. What may be the correct concentration for one may be too high for another. Review of literature sources showed that six molarity sodium hydroxide (6M NaOH) provided adequate etch rates for the lexan polycarbonate, cellulose nitrate, and CR-39. Therefore, 6M NaOH was used in all three etching procedures. The conductive liquid in the ECE process was a 10% sodium chloride (10% NaCl) solution.

V. Experimental Equipment

The experimental equipment used can be categorized into three main areas. These areas are: 1) equipment used to expose the track etch materials to alpha particles; 2) equipment used to etch the exposed materials; and 3) equipment used to observe the materials after etching. Table 1 provides a complete list of equipment by category. A general description of the major equipment in each area is provided in the following three sections.

Exposure Equipment

Three methods of alpha exposure were performed on the track etch materials. The methods were : 1) exposure to 5.47 Mev alpha particles from ^{241}Am; 2) exposure to alpha particles emitted by radon and its progeny; 3) exposure to alpha particles and lithium atoms from the $^{10}B(n,\alpha)^{7}Li$ reaction. The radon and $^{10}B(n,\alpha)^{7}Li$ exposure procedures are described briefly in the next two paragraphs.

Radon exposure was performed in a radon room set up by Captain David Little, U.S. Army, for his thesis project (14). Figure 9 shows a schematic of the room. Radon gas was generated and forced through plastic tubes on the floor of the test room. Tiny holes, which were drilled in the tubes at set intervals, allows the radon gas to escape and fill the room. Natural percolation of radon gas from soil is simulated in this manner.

Neutron exposure, to initiate the $^{10}B(n,\alpha)^{7}Li$ reaction, was performed using a thermal pile. Figure 10 shows a schematic of the thermal pile. The thermal pile consists of a PuBe source embedded in a

Table I

List of Experimental Equipment Used
by Category

Exposure Equipment

^{241}Am source (1.15×10^{5}dpm)

Radon Room

Thermal Pile

Etching Equipment

Chemical Etch Bath

ECE System

- ECE chamber
- ECE power supply
- oven
- rubber gaskets
- Lubriseal

Observation Equipment

Leitz Microscope

- 3x to 500x
- translation table

Video Camera System

- video camera
- black & white monitor
- video recorder

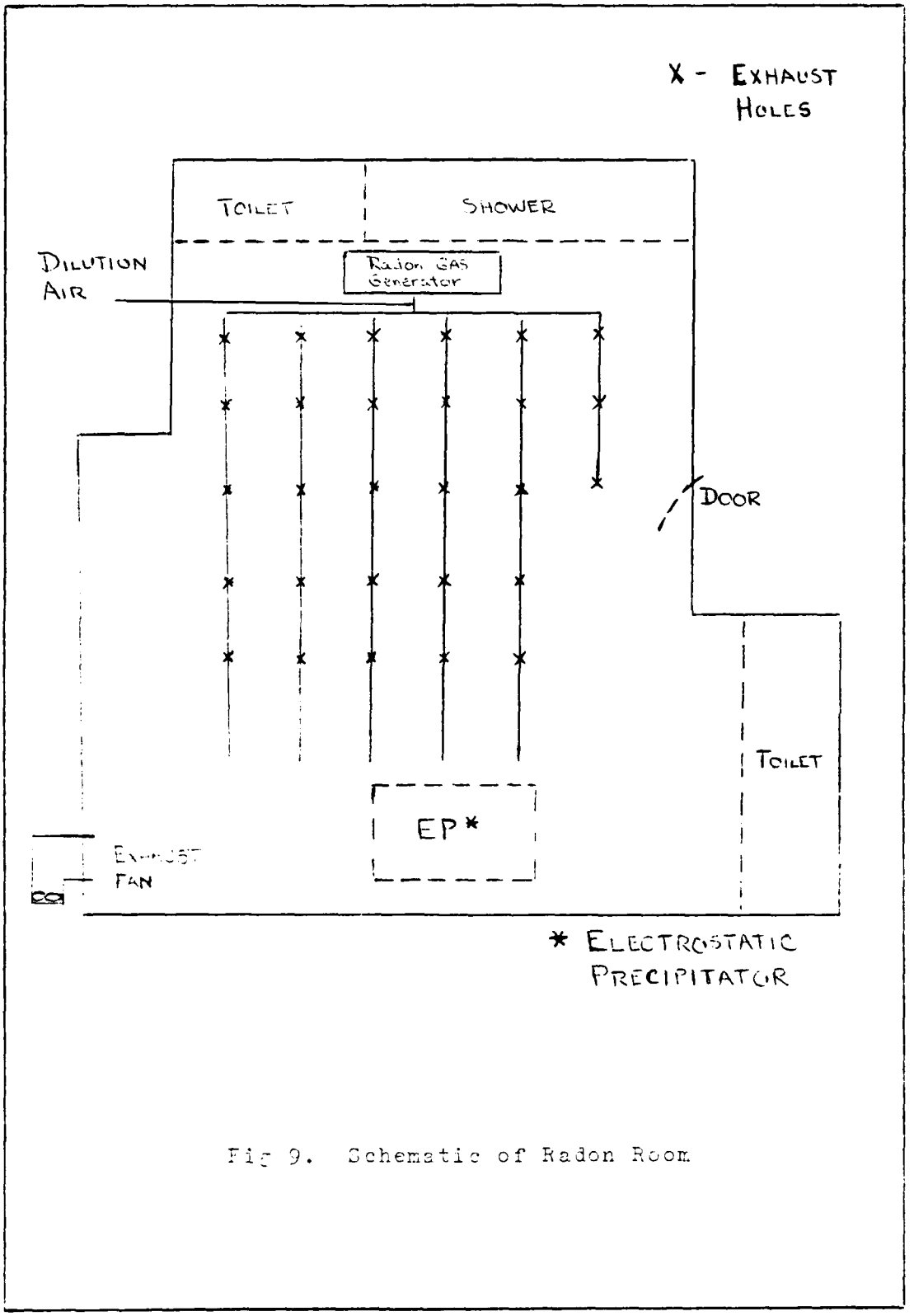

Fig 9. Schematic of Radon Room

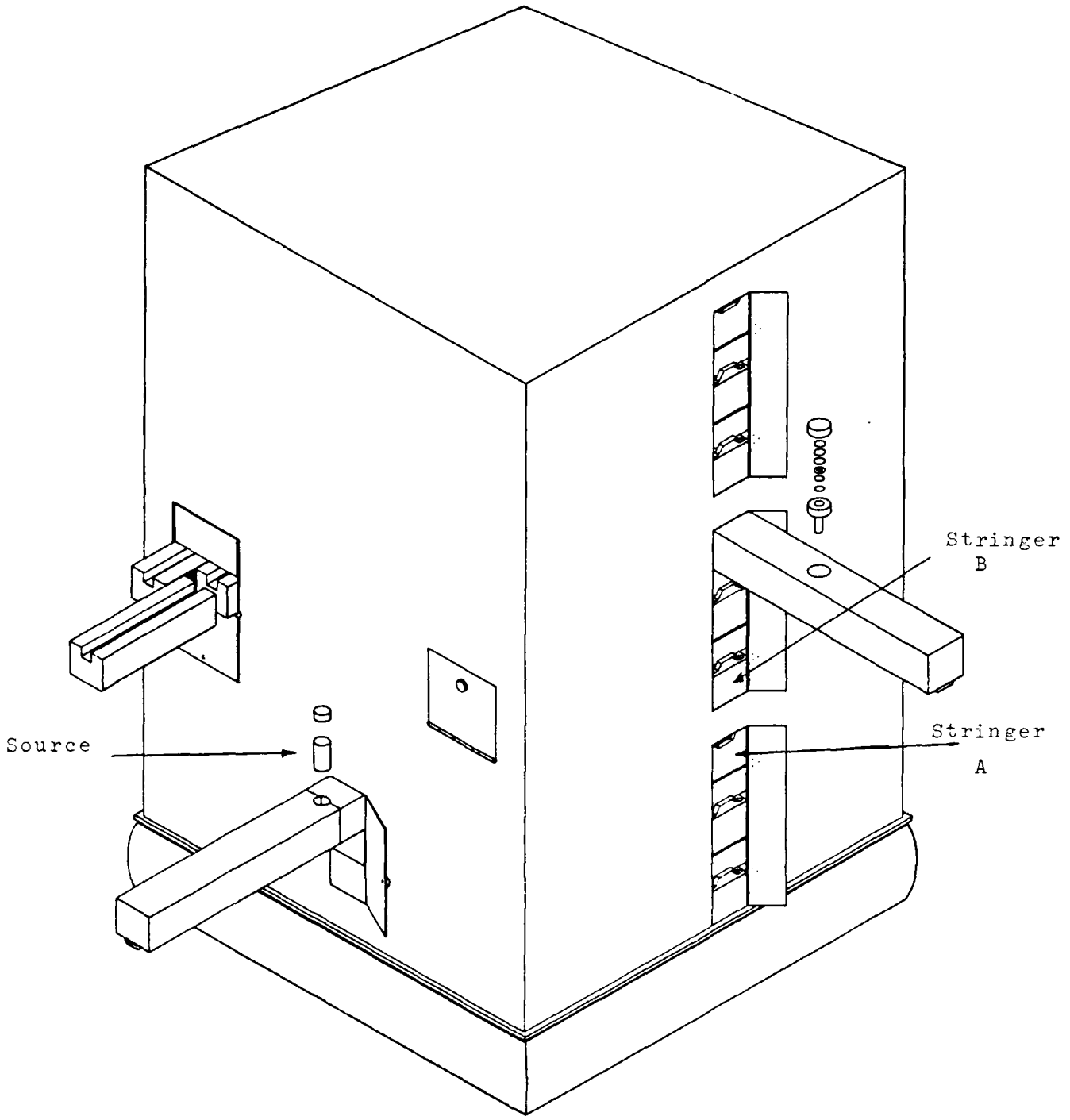

Fig 10. Schematic of Thermal Pile

cube of graphite. The PuBe source emits fast neutrons which are thermalized by interaction with the graphite. Materials to be irradiated are placed in one of two pre-cut openings, called stringers, in the graphite. Stringer A is 4.115 inches above the source and has a thermal neutron flux of 9600 neutrons per cm^2-sec. Stringer B is 12.115 inches above the source and has a thermal neutron flux of 8000 neutrons per cm^2-sec. The stringers are then returned to the pile and neutron irradiation begins.

Etching Equipment

Two types of etching equipment were designed and constructed. They were a chemical etch bath and an ECE system. The chemical etch bath is shown in Figure 11. Components of the chemical etch bath are: 1) a hot water bath; 2) a mixing and heating system; and 3) an etchant container. The hot water bath is in a stainless steel tank surrounded by styrofoam and plexiglass. The styrofoam insulates the tank so that a constant 60°C bath temperature can be maintained. The plexiglass top serves two purposes. It not only holds the mixing and heating system and the etchant container, but also reduces evaporation from the hot water bath. Evaporation is a major problem when etch times of several hours are performed. The mixing and heating system insures a constant 60°C temperature throughout the hot water bath. The etchant container is a 400 ml pyrex container in which the chemical etchant is held. The etchant container is also covered, by a rubber stopper, to eliminate etchant evaporation during long etch procedures. Exposed track etch materials are placed into the container and etched to prescribed time limits.

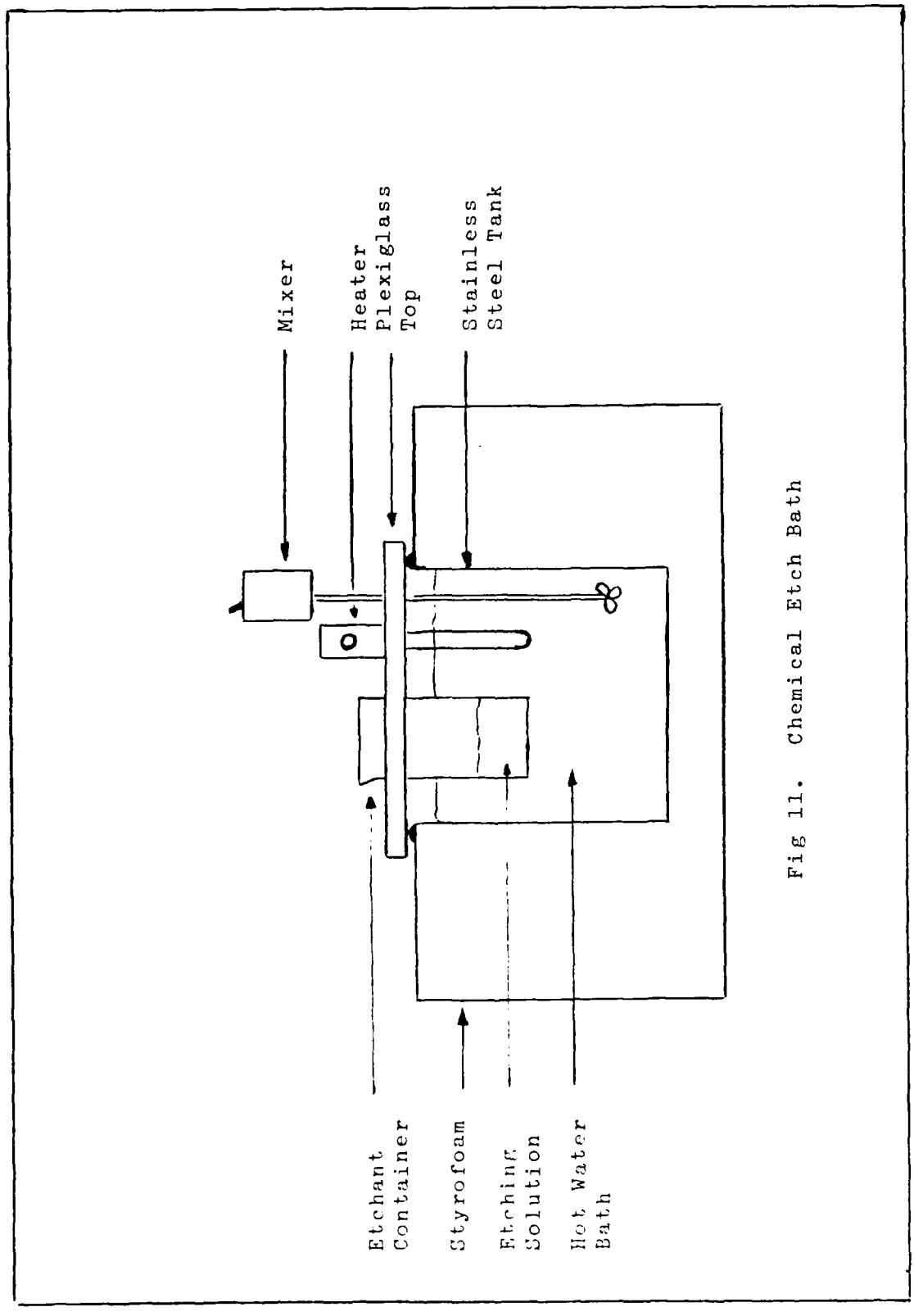

Fig 11. Chemical Etch Bath

The ECE equipment consists of an ECE chamber, a power supply, and a standard laboratory oven. Figure 12 provides a schematic of the ECE chamber. The ECE chamber consists of two plexiglass blocks in which circular reservoirs are drilled. Two holes, one large and one small, were then drilled into the sides of each reservoir. A stainless steel electrode was sealed in the small hole and the other remained open. Approximately 40 ml of chemical etchant and conductive liquid are poured into separate reservoirs through the large openings. The openings are then surrounded by a thin rubber gasket which is covered by a high pressure, high temperature sealant. Exposed track etch material is positioned between the gaskets and the block halves are joined together and held in place with C-clamps. The ECE chamber is turned upside down, allowing the solutions to come in contact with the track etch material. The sealant and rubber gaskets prevent leakage and mixing of the two solutions. The chamber is then placed in a 60°C oven and insulated wires, from the power supply, are attached to the electrodes. With no voltage applied, chemical etching is completed until one initiates ECE, at which time the AC power supply is turned on.

The power supply provides 0-1500 volts at a frequency of 60 Hz. Figure 13 shows a block diagram of this unit. The circuit was fused to automatically shut down if an electrical short occurred in the system. An electric clock was integrated into this fused area so that time of ECE exposure, before shorting, could be determined.

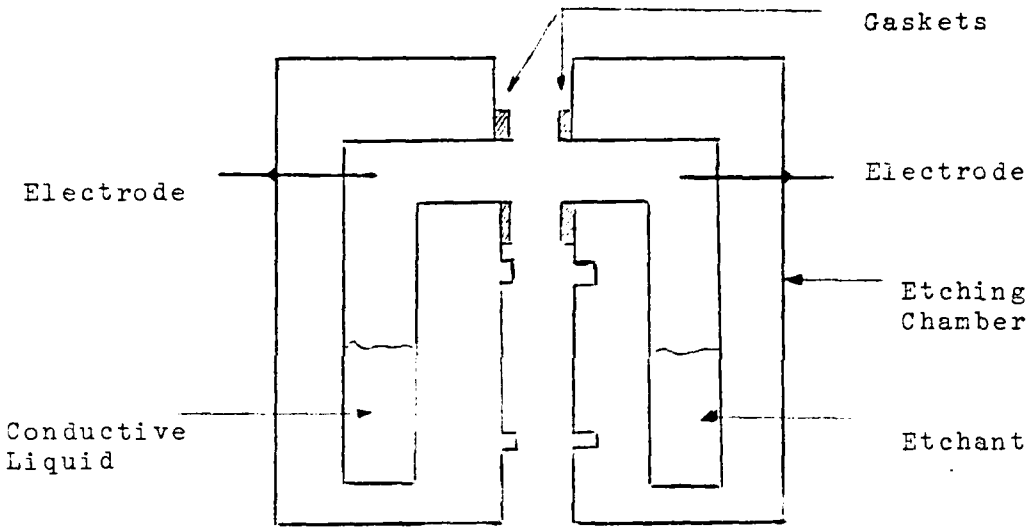

Fig 12. Electrochemical Etching Chamber

Observation Equipment

Tracks formed within the track etch materials were analyzed using a Leitz optical microscope under 3-500 power magnification. Figure 14 provides a drawing of the microscope and modifications made to it. The microscope was modified by the addition of a video camera system and translation table. The video system contained a camera, a black and white monitor, and a video recorder. The video camera was attached to the upper eye piece of the microscope. Output from the camera was transferred to the monitor and video recorder. In this manner, track etch materials could be viewed through the eyepieces or on the monitor. This system helped relieve eye strain caused by many hours of

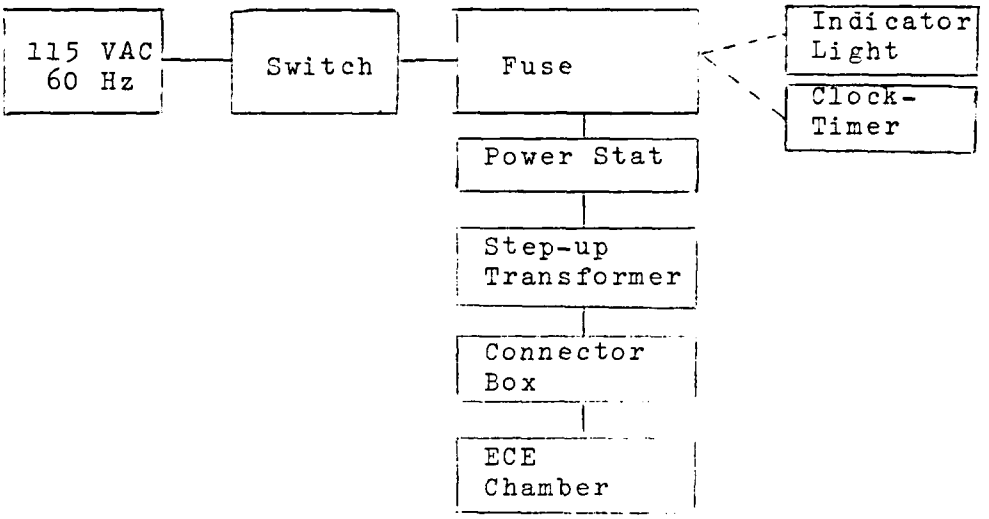

Fig 13. Block Diagram of Electrochemical Etching System Power Supply

prolonged viewing. It also provided enlargement and contrasting of the area under observation. This made distinguishing damage tracks from imperfections much easier. All observations were made using transmitted light which makes the damage tracks dark on a white background.

The translation table allowed the track etch materials to be moved horizontally and vertically in small increments. Each scale was numbered, permitting observation of prominent track formations to be categorized by X and Y location. Repetition of the observation could then be performed quickly and precisely.

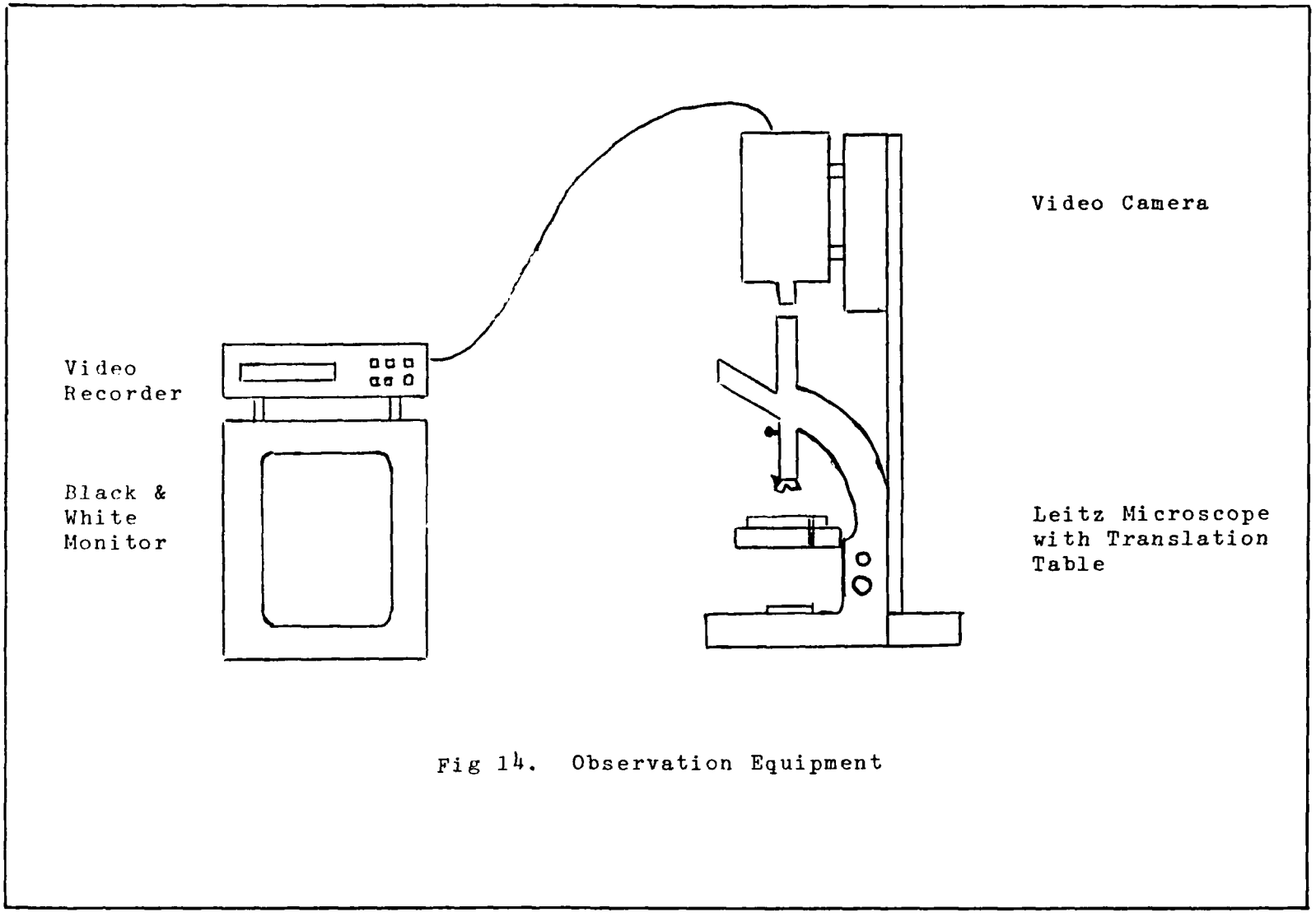

Fig 14. Observation Equipment

VI. Experimental Procedure and Results

Three track etch materials were exposed and etched. The materials were lexan polycarbonate, cellulose nitrate, and CR-39. This chapter is divided into three sections. Each section outlines the track etch material investigated, the exposure and etch procedures performed, and the qualitative observations made on the etched material.

Lexan Polycarbonate Exposure and Etch Procedures

The lexan polycarbonate track etch material was exposed to alpha particles by two methods. The first method was direct exposure to alphas from an ^{241}Am source. ^{241}Am emits alphas with an energy of 5.47 MeV. This energy is too high for lexan polycarbonate to register and therefore it, was lowered to 1.5 MeV by placing the source 2.5 cm (in air) above the lexan polycarbonate slide. The second method of exposure was by alpha particles and lithium atoms emitted from the $^{10}B(n,\alpha)^{7}Li$ reaction. The reaction was obtained by the following procedure. Amorphous boron was suspended in a flexible colloidian and then placed on a lexan polycarbonate slide. The colloidian was allowed to harden, placing the boron particles in intimate contact with the slide. The slide was then placed in one of two stringers in the thermal pile. Stringer A was 4.115 inches above the PuBe source and had a thermal neutron flux of 9600 neutrons per cm^2-sec. Stringer B was 12.115 inches above the PuBe source and had a thermal neutron flux of 8000 neutrons per cm^2-sec. The slide was irradiated for various time intervals between 24 and 240 hours.

The number of alpha particles and lithium atoms emitted by the $^{10}B(n,\alpha)^{7}Li$ reaction is calculated using the following formulation (4:173).

$$\text{Reaction Rate} = \sigma N \phi \qquad (5)$$

where

σ is the thermal neutron capture cross section in barns

N is the number density of target atoms present

ϕ is the thermal neutron flux in neutrons/cm^2-sec

$$N = \frac{m \delta Na}{A} \qquad (6)$$

where

m is the mass of the target material

δ is the isotopic concentration of the target material

Na is Avagadro's number (6.023×10^{23} atoms/gm-atom)

A is the mass number of the target material

In Eq 6 δ equals 19.6% and A equals 10.811 grams. The weight of boron added to the colloidian was always less than 0.01 grams. Placing this maximum limit for m in Eq 6 yields a value of 1.092×10^{20} ^{10}B atoms present at the time of irradiation. Substituting this value of N in Eq 5, and assuming a flux of 9600 neutrons per cm^2-sec, yields a maximum capture reaction rate of 4,022 reactions per second. Since every capture reaction releases one alpha particle and one lithium atom, then 4,022 alpha particles and 4,022 lithium atoms are emitted from the target area.

After irradiation, the slide was removed from the thermal pile and washed in methonal. The methanol bath dissolved the boron impregnated colloidian without harming the lexan slide's chemical composition or structure. Three slides were exposed in this manner. The slides were then etched by chemical etching, chemical pre-etching plus ECE, and ECE only. Electrochemical etching of lexan polycarbonate differs from the standard procedure discussed in Chapter IV. Lexan polycarbonate required etchant to be placed in both reservoirs of the ECE chamber, because of its poor alpha sensitivity. By placing etchant on both sides, surface imperfections are removed from the dielectric solid making track observation easier. The standard procedure required placement of etchant in one reservoir and a conductive liquid in the second.

Table 2 provides a tabulation of exposure method and times, etching method and times, and observations made on the various lexan polycarbonate slides.

Observations. All three methods of damage track enhancement

Table II

Exposure and Etch Procedures Performed
on Lexan Polycarbonate Slides

Slide	Exposure		Particles Incident on Slide ($\times 10^6$)	Etching		Observations
	Method	Time		Method	Time	
A1	^{241}Am	10 min	1.15	CE	3 hrs	Background hazed. Numerous tracks visible. Many are clustered together. Tracks are large and round in shape.
A2N	$^{10}B(n,\alpha)^{7}Li$	4 days	14.50	CE + ECE	45 min + 3 hrs	Background scratched but numerous tracks of various sizes are visible. Tracks are irregular in shape with "fingers" coming out of the center core.
A3N	$^{10}B(n,\alpha)^{7}Li$	1 day	7.96	ECE	3 hrs	Background somewhat marred but a number of various size tracks are visible. Tracks are similar to CE + ECE tracks.

CE - chemical etching at 60°

ECE- electrochemical etching at 60°C and 600 volts

performed well on the lexan polycarbonate slides. Figure 15 shows 1.5 MeV alpha particle tracks formed after chemical etching for 3 hours. The damage tracks are small, well rounded, and contrasted from the background imperfections. Figure 16 shows damage tracks formed in a slide exposed to the $^{10}B(n,\alpha)^{7}Li$ reaction. The slide was chemically pre-etched for three hours followed by ECE for three hours. The damage tracks are larger, irregular in shape, with "fingers" protruding from a central core. The central core is the primary damage track formed by the incident alpha particle or lithium atom. Electrochemical etching without chemical pre-etch resulted in similar track formation with fewer than expected tracks being visible.

Cellulose Nitrate Exposure and Etch Procedures

The cellulose nitrate film was manufactured in the radio-chemistry laboratory per instructions provided by Dr. Philip Jenkins of Mound Laboratory (15:55-77). The film was approximately 15 mils thick and therefore could not be electrochemically etched. The film was exposed to 4.5 MeV alphas from an ^{241}Am source. The 4.5 MeV alphas were obtained by placing the source 1 cm (in air) above the film. After exposure the film was chemically etched at room temperature and 60°C.

Exposure of the film to the $^{10}B(n,\alpha)^{7}Li$ was not performed due to the composition of the flexible colloidian used to suspend the boron particles. The flexible colloidian is a solution of cellulose nitrate suspended in ether. Once the colloidian hardened on the manufactured film, it could not be removed. Table 3 provides a tabulation of the exposure times, etching method and times, and qualitative observations made on the film.

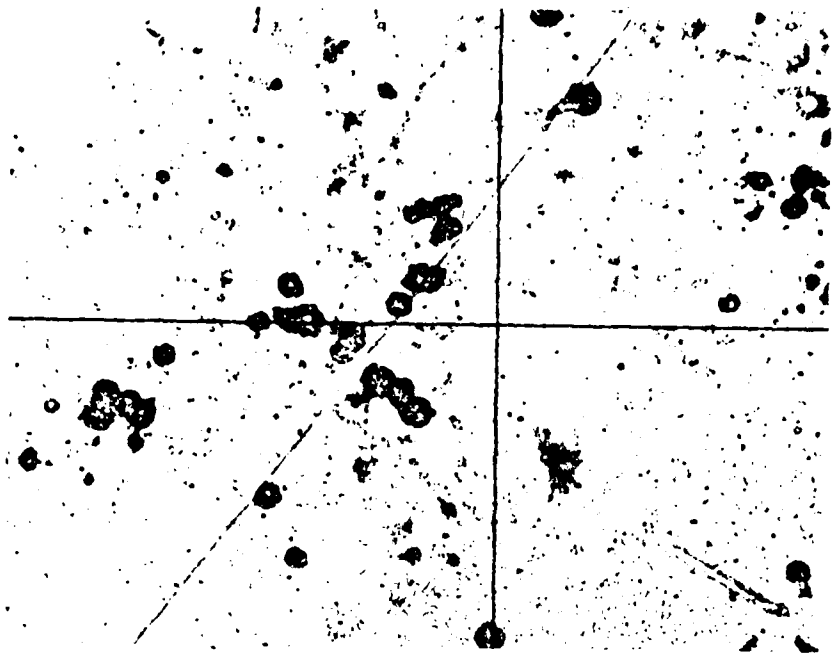

Fig 15. 1.5 Mev Alpha Tracks in Lexan Polycarbonate at 200x After Three Hours of Chemical Etching

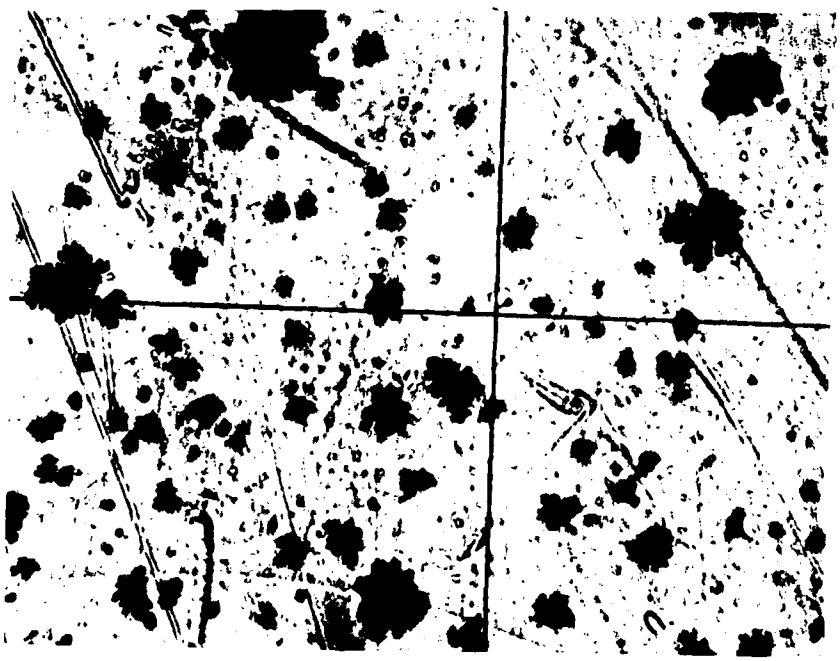

Fig 16. $^{10}B(n,\alpha)^{7}Li$ Tracks in Lexan Polycarbonate at 200x After Three Hours of Chemical Etching Followed by ECE for Three Hours

Table III

Exposure and Etch Procedures Performed
on Cellulose Nitrate Film

Slide	Exposure		Etching		Observations
	Method	Time	Method	Time	
1	4.5 MeV Alphas	10 min ***	CE (60)	5 min	Film was totally dissolved. Etchant smelled of banana oil.
2			CE (60)	3 min	Film was partially dissolved. Etchant smelled of banana oil.
3			CE (60)	2 min	Film intact. No tracks visible due to background imperfections.
4			CE (RT)	1 hr	Film intact. Background was somewhat clearer but still no tracks visible.
5			CE (RT)	3 hrs	Film intact. Background was the same as after one hour CE at room temperature.

CE (60) - chemical etching at 60°C
CE (RT) - chemical etching at room temperature
***Total number of particles incident on slides after 10 minutes is 1.15×10^6

Observations. All observations of the cellulose nitrate film were negative due to its poor manufactured quality. The film contained large numbers of surface and subsurface imperfections and dust particles. These imperfections made distinguishing damage tracks from background clutter impossible. This type of cellulose nitrate was therefore dropped from further investigation.

CR-39 Exposure and Etch Procedures

The CR-39 track etch material was exposed to alpha particles by three methods. These methods were : 1) exposure by alpha particles from an ^{241}Am source; 2) exposure by alpha particles and lithium atoms emitted from the $^{10}B(n,\alpha)^{7}Li$ reaction; and 3) exposure of alphas emitted from the decay of radon gas and its daughter products. Direct exposure was performed by placing an ^{241}Am source directly on the CR-39 slide. This resulted in 5.47 MeV alpha particles incident on the surface of the slide. Exposure from the $^{10}B(n,\alpha)^{7}Li$ reaction was the same as performed on the lexan polycarbonate slides. Exposure by alphas emitted from the decay of radon and its daughter products was performed in a radon room described in Chapter V. Several CR-39 slides were placed at various locations within the room. The slides were exposed to the radon and daughter products for a period of two to four weeks. The long exposure times were required because of the relatively "low" alpha emission rate.

The exposed slides were then etched by CE, ECE and their combination. Table 4 provides a tabulation of exposure method and times, etching methods and times, and qualitative observations made on the slides.

Table IV

Exposure and Etch Procedures Performed
on CR-39 Slides

Slide	Exposure		Particles Incident on slide ($x10^6$)	Etching		Observations
	Method	Time		Method	Time	
B1	Radon + daughters	2 wks	?	ECE	3 hrs	Background filled with scratch marks but numerous tracks visible. Tracks are small, irregular in shape with "fingers" protruding from center.
B2	Radon + daughters	2 wks	?	CE	3 hrs	Background clear but no tracks visible.
B4	^{241}Am*	10 min	1.15	CE	2 hrs	Background clear. Only a few tracks visible under 500X.
B5	^{241}Am**	10 min	1.15	CE	4 hrs	Background clear with thousands of small tracks visible. Tracks stop almost immediately outside marker ring. (Marker ring equals dimension of ^{241}Am source)

CE - chemical etching at 60°C

ECE- electrochemical etching at 60°C and 1380 volts

Table IV (continued)

Exposure and Etch Procedures
Performed on CR-39 Slides

Slide	Exposure		Particles Incident on slide ($x10^6$)	Etching		Observations
	Method	Time		Method	Time	
B3N	$^{10}B(n,\alpha)^{7}Li$	10 days	13.3	CE	1 hr	Background has a large number of scratch marks. Area that was covered by boron is almost one continuous track at 500X. Tracks stop once outside region covered by boron.
B3NN	$^{10}B(n,\alpha)^{7}Li$	1 day	4.4	ECE	3 hrs	Background has a large number of scratch marks. A small number of tracks are visible. The tracks are fairly large and irregular in shape with "fingers" coming from the center core.
B4N	$^{10}B(n,\alpha)^{7}Li$	1 day	2.5	CE + ECE	3 hrs + 3 hrs	Background is a little clearer. Numerous tracks are visible. The tracks are large and irregular in shape with "fingers" coming from the center core.

CE - chemical etching at 60°C

ECE- electrochemical etching at 60°C and 1380 volts

Observations. Chemical etching and chemical pre-etching followed by ECE provided the best damage track formation. Figure 17 shows 5.47 MeV alpha particle tracks enhanced by chemical etching for three hours. The damage tracks are fairly large, well rounded, and contrasted from the background imperfections. Figure 18 shows tracks formed in a CR-39 slide exposed to alpha particles and lithium atoms, from the $^{10}B(n,\alpha)^{7}Li$ reaction. The slide was chemically etched for three hours, followed by three hours of ECE at 60°C and 1380 volts. The damage tracks are larger, irregular in shape, with "fingers" protruding from a central core. These tracks are similar to those observed in the lexan polycarbonate slides under the same etch conditions. Electrochemical etching with no pre-etch produced the same type of tracks, but fewer in number. All other exposure and etch combinations resulted in the formation of one of these two types of tracks.

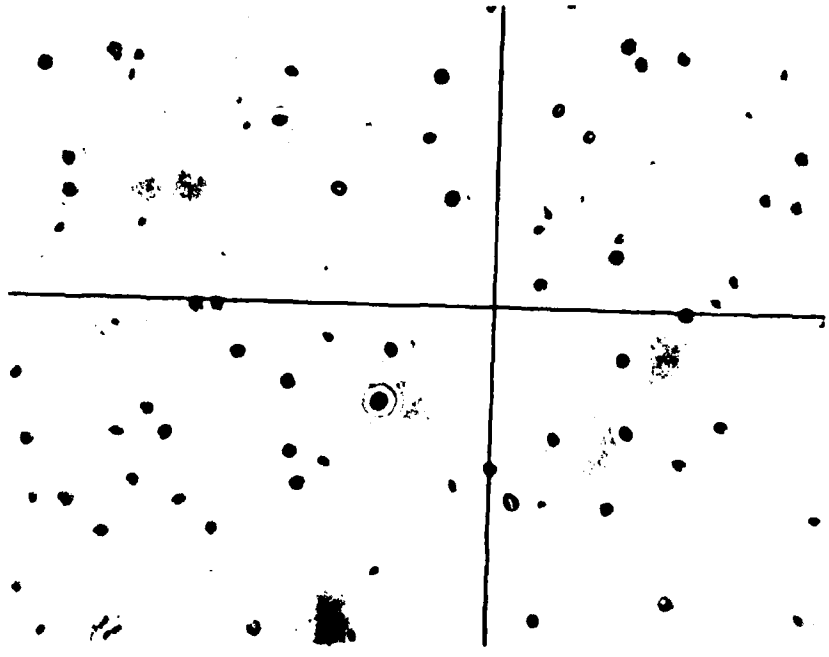

Fig 17. 5.47 Mev Alpha Tracks in CR-39 at 500x After Three Hours of Chemical Etching

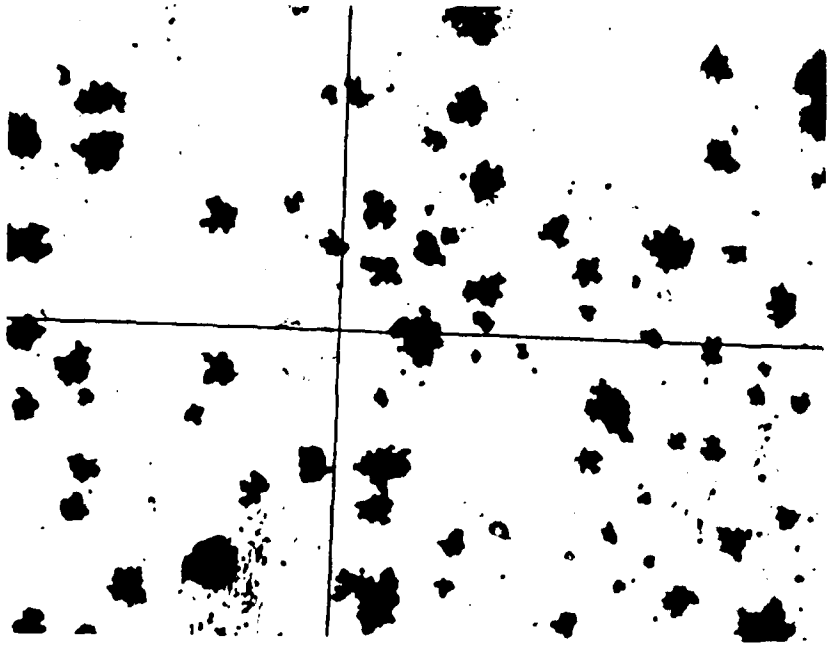

Fig 18. $^{10}B(n,\alpha)^{7}Li$ Tracks in CR-39 at 200x After Three Hours of Chemical Etching Followed by ECE for Three Hours

VII. Summary and Recommendations

Summary

The objective of this study was to compile and perform track etch procedures capable of detecting alpha particle interactions within track etch materials. Three track etch materials were chosen for this investigation because of either their present use in the environmental studies field or "high" alpha sensitivity. The materials chosen were Lexan polycarbonate, cellulose nitrate, and CR-39. The materials were exposed to a variety of alpha interactions and then etched by three methods. The methods were chemical etching, chemical pre-etching followed by electrochemical etching (ECE), and electrochemical etching only. The lexan polycarbonate and CR-39 performed well under these etching procedures and produced two types of tracks. The type of tracks formed was related to the etching procedure used. Chemical etching produced a fairly large, well rounded hole, which contrasted from the background imperfections. Chemical pre-etching followed by ECE and pure ECE only formed tracks that were larger and irregular in size with "fingers" protruding from a central core. The cellulose nitrate track etch material was dropped from further analysis because of its poor manufactured quality. This poor quality made distinguishing damage tracks from background imperfections impossible. The results showed that detection of low energy alpha particles ($\leq$2.0 Mev), was possible using lexan polycarbonate and the etching procedures. They also showed that detection of alpha particles ($\leq$8 Mev) was possible using CR-39 and the three etching procedures. Thus

detection of low energy alpha particles, using track etch materials and etching procedures, is viable.

Recommendations

Based on observations and problems encountered during the study, the following recommendations are proposed for further investigation:

1. Although the study provided basic procedures for etching track etch materials, it did not fully optimize them. Further investigation should be performed to optimize each material's track enhancement by varying the: a) etchant concentration; b) etchant viscosity; c) etchant temperature; and d) etching time.
2. Damage track size is dependent on the fluence and energy of the particle incident on the dielectric solid. One should determine the proportionality between damage track size and these factors. This can be performed by exposing the track etch materials to alpha particles of various energy and fluence and then chemically etching the slides for a standard time limit. Measuring the various size tracks produced, under the different exposure conditions, will provide the desired proportionality factors.
3. Because of the poor quality of the manufactured cellulose nitrate films, observation of alpha particle interactions was not possible. The exposure and etch procedures should be repeated using LR-115. LR-115 is a cellulose nitrate film manufactured by Kodak-Pathe of France. It is suitably pure for use as a track etch detector.
4. As now designed, the chemical etch bath can effectively etch one slide, in one etch solution at a time. The bath should be modified so several slides can be etched at one time with each in

a different solution. This can be completed by modifying the plexiglass top to hold several individual large test tubes instead of a single 400 ml pyrex beaker.

5. During the first attempts of electrochemical etching, numerous leaks caused the power system to short out. Modification of the chamber by adding rubber gaskets and lubriseal stopped the leakage, but was extremely messy and ineffective. The chamber should be modified so the track etch materials are securely held in place between two O-ring seals. The O-ring seals will prevent the solutions from leaking and causing a short in the power supply.

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Appendix

Track Formation Theory

Possible track production mechanisms are different for the two types of track etch materials (i.e. organic and inorganic). General track formation theory states that there are two main factors which produce damage tracks. These factors are primary excitation and ionization caused by the charged particle and secondary excitation and ionization caused by the delta rays. In the case of inorganic detectors, the secondary excitation and ionization damage caused by the delta rays can be neglected. The most probable track production mechanisms in organic solids are secondary energy loss and restricted energy loss.

In organic solids damage along the track consists mainly of displaced atoms. Previous research has shown that the atoms are not displaced by direct atomic collision, but by interaction with the electrons in the atom. The ion explosion spike theory was hypothesized to explain this discrepancy. In this theory, passage of the positively charged particle creates an electrostatically unstable formation of adjacent positive ions by the stripping off of electrons. These unstable ions then repel each other and they are ejected from their normal locations into interstitial positions (1:32). This electrostatic instability causes local stresses to develop at the vacancy and interstitial sites. The local stresses are then spread throughout a larger region, surrounding the vacancy and interstitial sites, by elastic relaxation. It is this spreading of the stresses which forms

the damage track. Criteria for the initiation of this process is that the local electrostatic stress be greater than the atomic bonding energy. Figure 19 shows this three-step process.

Two other theories deal specifically with organic polymers. Secondary ionization and excitation effects are prime factors in damage track formation in these materials. Each theory will be described briefly. In the specific energy loss theory, track formation can only occur when a critical dose of energy is deposited in a specific radial distance of the incident charged particle path. The critical dose of energy breaks long chain polymer bonds to make track enhancement possible. This critical dose, E (energy per unit volume), and radial distance are determined experimentally for different organic polymers.

Problems with this theory are that it: 1) ignores defects produced by primary excitation and ionization; 2) extrapolates the critical energy incorrectly at low energies; and 3) does not predict the relative thresholds or absence of track formation in conductors (1:40).

In the restricted energy loss theory, damage tracks are formed only by that part of the primary ionization and excitation energy lost in accelerating moderately low energy delta rays (1:38).

Problems with this theory are that it: 1) ignores interior dose deposited by the delta rays outside the etchable region; 2) predicts track registration where not experimentally observed; 3) ignores the qualitatively different defects formed by the primary and secondary ionization and excitation events; and 4) does not predict relative thresholds or absence of track formation in conductors (1:40).

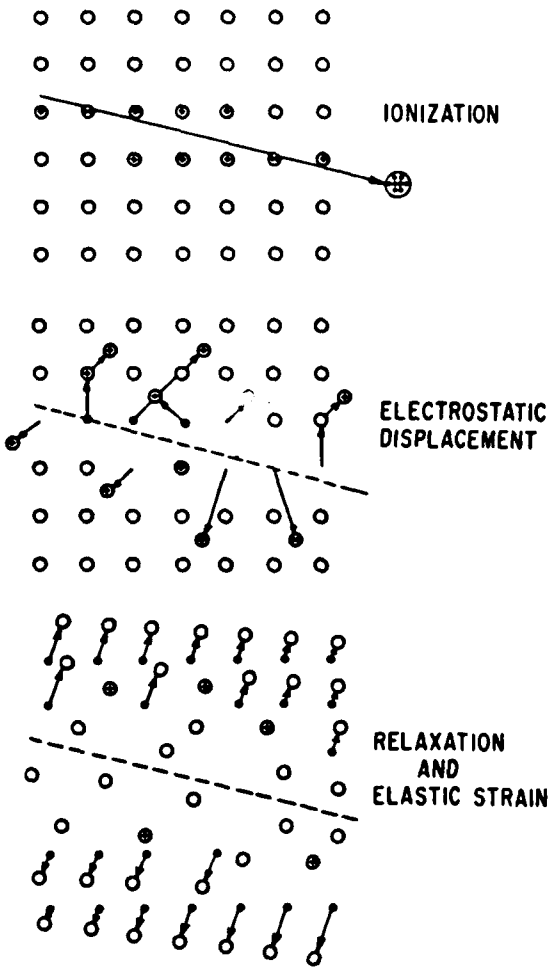

Fig. 19 Ion Explosion Spike Track Formation Mechanism

Other theories of track formation in organic and inorganic solids have been suggested. However, they contain more problem areas than those presented and were deleted from further consideration.

VITA

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In this study I have developed and set up experimental procedures and methodologies for detection of low energy alpha particles using the track etch technique. Three track etch materials, lexan polycarbonate, cellulose nitrate, and CR-39, were exposed to various energy alpha particles. The exposure resulted in the formation of atomic scale damage tracks within each material. Because the damage tracks were on an atomic scale in size, they could not be optically seen unless enhanced or enlarged by some physical means. Three methods of enhancement were performed on the exposed track etch materials. These methods were chemical etching, electrochemical etching, and a combination of the two. The results indicted that all three track etching methods provided adequate enhancement of the alpha damage tracks, thereby showing that low energy charged particles can be detected using the track etch technique.

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